

MERCURY IN SOLID OR SEMISOLID WASTE (MANUAL COLD-VAPOR TECHNIQUE) EPA 7471B REVISION 2 2007					
Facility Name: _____ VELAP ID _____					
Assessor Name: _____ Analyst Name: _____ Inspection Date _____					
Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
Was aqua regia prepared immediately prior to use with three parts HCl to one part HNO <sub>3</sub> ?	7.3				
Were samples held for not longer than 28 days prior to analysis?	8.3				
Were solid samples refrigerated until analysis?	8.3				
Was at least one method blank analyzed with each batch of samples processed?	9.3				
Were laboratory control samples carried through all aspects of processing and analysis and analyzed to be within $\pm 20\%$ or within historical compiled control limits?	9.4				
Were laboratory control samples analyzed every ten samples?	9.4				
If method blanks or laboratory control samples were analyzed to be not acceptable, were they re-run only once more?	9.3, 9.4				
If method blanks or laboratory control samples failed second analyses, were all the samples from the previous passing LCS or blank reprepared and reanalyzed?	9.4				
Were a spike and a duplicate or spike duplicate prepared and analyzed with each batch, with an acceptance range within $\pm 20\%$ ?	9.5				
Were 5 mL aliquots of aqua regia added to 10 mL volumes of calibration standards, and the solutions heated for 2 minutes at $95 \pm 3^\circ\text{C}$ ?	10.1				
Were 50 mL of reagent water and 15 mL of potassium permanganate then added to each bottle?	10.1				
Were the bottles reheated at $95 \pm 3^\circ\text{C}$ for 30 minutes?	10.1				
Were bottles then cooled and 6 mL portions of sodium chloride-hydroxylamine sulfate added?	10.1				
Notes/Comments:					

<b>MERCURY IN SOLID OR SEMISOLID WASTE (MANUAL COLD-VAPOR TECHNIQUE)</b> <b>EPA 7471B REVISION 2 2007</b>					
<b>Relevant Aspect of Standards</b>	<b>Method Reference</b>	<b>Y</b>	<b>N</b>	<b>N/A</b>	<b>Comments</b>
Were 50 mL portions of reagent water then added to the bottles, followed by 5 mL of stannous sulfate solution? (10% stannous chloride may be substituted per section 7.5.)	10.1				
Were calibration standards then analyzed and their absorbances plotted versus micrograms of mercury?	10.2				
Were 5 mL of reagent water and 5 mL of aqua regia added to weighed portions of sample?	11.1				
Were sample digestion containers heated at $95 \pm 3^{\circ}\text{C}$ for 2 minutes?	11.1				
Were 50 mL of reagent water and 15 mL of potassium permanganate then added to each sample digest?	11.1				
Did purple color persist for at least 15 minutes?	11.1				
If purple color did not persist for at least 15 minutes, were additional portions of permanganate added?	11.1				
Were the same portions of permanganate added to all samples, standards, and QC in a batch?	11.1				
Were digests heated at $95 \pm 3^{\circ}\text{C}$ for 30 minutes?	11.1				
Were digests cooled, 6 mL portions of sodium chloride-hydroxylamine solution added, and then 5 mL portions of stannous sulfate added?	11.1				
<b>Alternate Digestion Procedure:</b>					
Were 5 mL of concentrated $\text{H}_2\text{SO}_4$ , 2 mL of concentrated $\text{HNO}_3$ , and 5 mL of saturated $\text{KMnO}_4$ added to each weighed sample?	11.2				
Were the digestion containers then covered with a piece of foil and autoclave at $121 \pm 3^{\circ}\text{C}$ and 15 lb for 15 minutes?	11.2				
Were the digested samples cooled and diluted to 100 mL with reagent water?	11.2				
Were the dead air spaces purged from the digestion containers?	11.2				
Were 6 mL portions of sodium chloride-hydroxylamine solution added to digestion containers?	11.1				
Were 5 mL portions of stannous sulfate added solutions to each digestion container?	11.1				
Notes/Comments:					